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AUTHOR(S):

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Prof
YOKO, Toshinobu
(D Eng)



Assoc Prof
TAKAHASHI, Masahide
(D Sc)



Assist Prof
TOKUDA, Yomei
(D Eng)



A Res
MENAA, Bouzid
(Ph D)



PD
KANG, Eun-Seok
(Ph D)

Students

MIZUNO, Megumi (D3)
OHTANI, Shoichi (M2)
MAEDA, Takahiro (M2)
MIYAMOTO, Ayako (M2)
KOUNO, Ryo (M1)

MATSUDA, Kazuomi (M1)
SUZUKI, Masaru (M1)
FUJII, Masao (UG)
UEMURA, Kouji (UG)
YAMAMOTO, Hideaki (UG)

FUKUDA, Masahiro (RF)
FUKUDA, Masaaki (RF)
KUNIYOSHI, Minoru (RF)
TOMOYOSHI, Yoshio (RF)
NAKATA, Kunihiko (RF)

Visitors

Prof INNOCENZI, Plinio
Prof BABONNEAU, Florence
Prof BONHOMME, Christian

University of Sassari, Italy, 17 January - 14 February 2005
Université Paris 6, France, 7 November 2005
Université Paris 6, France, 7 November 2005

Scope of Research

In this laboratory, amorphous and polycrystalline inorganic materials and organic-inorganic hybrid materials with various optical functions such as photorefractivity, optical nonlinearity and photocatalysis are the target materials, which are synthesized by sol-gel, multi-cathode sputtering, melt-quenching and sintering methods and so on. Aiming at highly functional materials the structures are investigated by X-ray diffraction techniques, high-resolution NMR, thermal analysis, various laser spectroscopies and ab initio molecular orbital calculations.

Research Activities (Year 2005)

Presentations

Optical Characteristics of Organic-inorganic Hybrid Material Derived Through Non-hydrolytic Reaction and Photo-Polymerization, Kang E., Takahashi M., Yoko T., The 105th Annual Meeting the American Ceramics Society, Baltimore, 15 - 17 April.

Preparation and NLO Properties of Dye-doped Polysiloxane-based Glass Thick Films, Menaa B., Takahashi M., Mizuno M., Tokuda Y., Yoko T., MC7: Functional Materials for the 21st Century, Edinburgh (UK), 6 - 9 July.

Organic-inorganic Hybrid Materials Prepared through Non-aqueous Acid-base Reactions, Takahashi M., Mizuno M., Kakiuchida H., Menaa B., Tokuda Y., Yoko T., 13th International Workshop on Sol-gel Science and Technology (Sol-gel2005), Los Angeles, USA, 21 - 26 September.

Optical Characteristics of Organic-inorganic Hybrid Material Derived through Non-hydrolytic Reaction and Photo-polymerization, Kang E., Takahashi M., Yoko T., 6th Pacificrim Conference on Ceramics and Glass Tech-

nology, Maui, Hawaii, USA, 16 September.

Grants

Yoko T., Preparation of Organic-inorganic Hybrid Low-melting Glasses through Acid-base Reaction, Asahi Glass Foundation, 1 April 2004 - 31 March 2005.

Takahashi M., Inhomogeneous Structures in the Glasses, Grant-in-Aid for Scientific Research for Encouragement of Young Scientists (A), 1 April 2004 - 31 March 2006.

Takahashi M., Development of Photonics Materials Based on the Organic-inorganic Hybrid Low Melting Glasses, PRESTO, Japan Science and Technology Agency, 1 November 2002 - 31 March 2006.

Takahashi M., Organic-inorganic Hybrid Low-melting Glasses Doped with Optical Active Centers via Non-aqueous Acid-base Reaction, Murata Scientific Foundation, 1 July 2004 - 31 June 2005.

Takahashi M., Fabrication of Large Area Photonic Films, Toyota Physical & Chemical Research Institute, 1 April

Fabrication of Periodic Photonic Structure of TiO₂ and Other Oxides on Sol-gel Dip Coated Films through Photo-Polymerization Induced Phase Separation (PIPS)

By combining PIPS with sol-gel coating technique, we can obtain photonic structures shown in Fig. 1. Pictures show the TiO₂ phase grating obtained by holographic illumination of Ar⁺-ion laser light (inset shows the SEM image of the obtained gratings). The periodicity could be controlled in the range from 500 nm to 20 μ m depending on the holographic condition of irradiated laser light. When the PIPS and sol-gel conditions are appropriately controlled, we can fabricate periodic structure without laser (coherent) light source. Fig. 2 shows an AFM image of the TiO₂ 2D-photonic structure obtained by the present method using a black light as UV source. The 2D structure is self-organized on the substrate. This method has a great advantage compared to the photo-polymerization method of vinyl-modified metal alkoxides system. With complete condensation, a large value of Δn is expected.

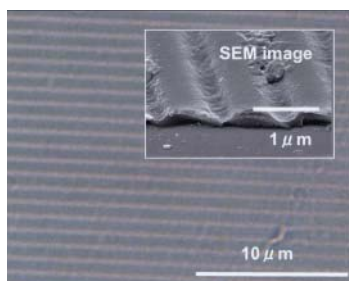


Figure 1. Titania phase grating obtained by PIPS in combination with the sol-gel coating technique. Inset shows the SEM image of fractured edge of the obtained grating.

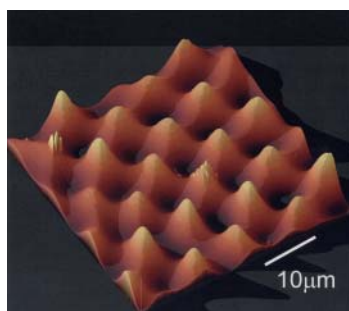


Figure 2. 2D photonic structure of TiO₂ obtained by irradiating a black light. The periodic structure was obtained by controlling PIPS conditions.

Structural Study on Organically-Modified Polysiloxane Glasses

We have already reported that low-melting glass can be prepared using a gel derived by the sol-gel method. The glasses with compositions of $x\text{Ph}_2\text{SiO}_{2/2}-(1-x)\text{PhSiO}_{3/2}$ ($0 < x \leq 30$) were prepared and their softening temperatures are widely dispersed around 150°C. In order to examine factors affecting the softening behavior, structural study has been performed using ²⁹Si MAS NMR (Magic Angle Spinning Nuclear Magnetic Resonance) spectroscopy and GPC (Gel Permeation Chromatography) measurements. First, we obtained the frequency at G'' (the imaginary part of the elastic modulus) = 10³ Pa using viscoelastic measurements. Second, we acquired the condensation degree of Si, $\langle m \rangle$, which is the number of Si-O-Si bonding per one Si by NMR spectra and the molecular volume, $\langle M \rangle$, by GPC measurements. Finally, we have found a relation between the frequency at $G''=10^3$ Pa, the condensation-degree and molecular volume as follows,

$$\log \omega_{G''=10^3 \text{ Pa}} = -9.2 \langle m \rangle - 5.0 \log \langle M \rangle + \text{const.}$$

We have also found that the rate of increase in elastic modulus induced by heat-treatment decreases with increasing amount of Ph₂SiO_{2/2} unit.

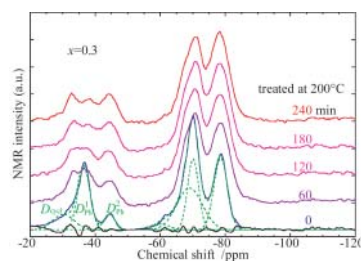


Figure 3a. ²⁹Si MAS NMR spectra of $x\text{Ph}_2\text{SiO}_{2/2}-(1-x)\text{PhSiO}_{3/2}$ glasses. Numericals represent the heat-treatment time.

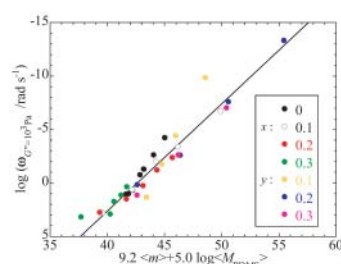


Figure 3b. A relationship between the frequency at $G''=10^3$ Pa, the condensation-degree and molecular volume for $x\text{Ph}_2\text{SiO}_{2/2}-(1-x)\text{PhSiO}_{3/2}$ glasses.

2005 - 31 March 2006.

Kakiuchida H., Softening Behavior of Organic-inorganic Hybrid Glasses and its Application for Photonic Devices, ICR Grants for Young Scientists.

Awards

6th Pacificrim Conference on Ceramics and Glass Technology, Student Poster Award, "Fabrication of TiO₂ Periodic Structure by the Photopolymerization-induced Phase Separation Method", Maeda T., Takahashi M., Yao J.,

Tokuda Y., Nishii J. and Yoko T., 6th Pacific Rim Conference on Ceramic and Glass Technology, 11 - 16 September 2005.

BCSJ Paper Award, "Conducting and Magnetic Properties of 1-Ethyl-3-methylimidazolium (EMI) Salts Containing Paramagnetic Irons: Liquids [EMI][M^{III}Cl₄] (M = Fe and Fe_{0.5}Ga_{0.5}) and Solid [EMI]₂[Fe^{II}Cl₄]", Yoshida Y., Otsuka A., Saito G., Natsume S., Nishibori E., Takata M., Sakata M., Takahashi M., and Yoko T.